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Chalcogenide glasses and glass-ceramics: Transparent materials in the infrared for dual applications

Verres et vitrocéramiques de chalcogénures : des matériaux transparents dans l'infrarouge pour des applications duales

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A B S T R A C T

In this paper are described the different research activities that led to the awarding of the Lamb prize by the French Academy of Sciences in order to promote research work on the national defense of France. This research concerns the development of infrared materials for night vision and the development of thermal imagers useful for defense, but also for civilian applications. The contribution has been particularly innovative in different sectors: broadening of chalcogenide glasses window of transparency, IR glass-ceramics with high thermomechanical properties, and the design of a new way of synthesis of these materials by a mechanical process.

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R É S U M É

Dans cet article sont décrites les activités de recherche qui ont permis l'obtention du prix Lamb, décerné par l'Académie des sciences dans le but de favoriser les études concernant la défense nationale de la France. Ces recherches concernent le développement de matériaux infrarouges pour la vision nocturne et la mise au point d'imageurs thermiques utiles pour des applications de défense, mais aussi civiles. La contribution est particulièrement innovante dans trois secteurs : l'élargissement du domaine spectral des verres de chalcogénures, les vitrocéramiques infrarouges à hautes propriétés thermomécaniques, ainsi que la conception d'une nouvelle voie de synthèse de ces matériaux par un procédé mécanique.

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1. Introduction

The main interest of chalcogenide glasses lies in their wide range of transparency, which can extend from the visible to the mid- infrared as a function of the chemical composition of the glass. Thus, the first part of the paper will be devoted to studying the extension of the windows of transparency of these materials in the visible or in the infrared domain. This research is mainly focused on obtaining multi-band materials, transparent in the visible range, but also in the atmospheric transparency band I (3–5 μm) and band II (8–12 μm).

Unlike many materials currently used for mid-infrared optical applications (monocrystalline Ge, polycrystalline ZnSe), chalcogenide glasses have a great advantage since they can be molded by simple heating above their glass transition temperature (T_g). Indeed, monocrystalline germanium and polycrystalline ZnSe require shaping by polishing at the diamond tip, a long and expensive process. Moreover, the relatively easy synthesis of chalcogenide glasses allows a reduction in the production costs of IR optics. These strengths have recently led to various infrared optical applications such as night vision, detection of chemical and biological species, production of infrared fibers or nonlinear optics... [1].

Nevertheless, chalcogenide glasses possess mechanical properties (hardness, toughness, etc.) which are relatively low compared to mono- or polycrystals (Ge, ZnSe...) regularly used in the infrared industry, limiting their fields of application. In the last works carried out by our team, we have demonstrated that these properties can be considerably improved by generating particles of nanometric to micronic size by appropriate heat treatment. The details of this work will be discussed later in this paper.

Furthermore, at the present time, the synthesis of chalcogenide glasses is carried out in single-use vacuum-sealed silica ampules. Thus, although the synthesis process is simple to implement, the fact that it is not possible to reuse the silica ampules and that many subsidiary steps of material recovery have to be carried out leads to an approximate increase of 30% in the price of the final glass, which makes them less competitive on the public market.

In order to make these infrared technologies accessible to civilians, it is therefore essential to develop new methods of synthesis at lower cost. Recent research project introduces an innovative way of synthesis, the first of which has been recently developed in the laboratory and will be described in the last section. This way of synthesis combining mechanical alloying and flash sintering should eventually open new routes to manufacture optical glasses, glass-ceramics, and ceramics.

2. Presentation of chalcogenide glasses

Numerous works and documents summarize the different stages of synthesis of pure chalcogenide glasses [2,3]. Although the synthesis process is identical, the shaping of the glasses may vary according to the applications envisaged (manufacture of lenses, fibers, thin films, etc.). Here will be recalled the main steps necessary to obtain a glass of chalcogenides using the conventional way of melting–quenching in vacuum-sealed silica ampules.

The elements necessary for the constitution of the glass are weighed in stoichiometric proportion before being placed in a silica tube. The silica tube will subsequently be sealed in a secondary vacuum. The sealed tube under vacuum is then heated in a rotating furnace at a temperature ranging from 700 °C to 950 °C, depending on the prepared composition. A phase of homogenization of the molten bath is then carried out before quenching the mixture in water or with compressed air.

The glass then undergoes an annealing treatment at a temperature slightly lower than the glass transition temperature T_g in order to relax the internal stresses caused during quenching. The bulk glass obtained can then be shaped (cutting, polishing, etc.) according to the targeted applications. In general, rods of 7 to 10 g having a diameter from 9 to 10 mm are synthesized, that is to say a height of about 6 cm.

Glass rods are then cut into 2-mm-thick slices and are then optically polished. In order to generate crystalline nanoparticles within the amorphous matrix, glasses will be annealed at about $T_g + 30\,^\circ\mathrm{C}$ for different times, leading to glass-ceramics that keep a wide range of transparency in the infrared range.

3. Broadening of the window of transparency

As mentioned above, the chalcogenide glasses possess a wide window of transparency in the infrared range covering the second (band I) and third (band II) atmospheric window between 3 to 5 μm and 8 to 12 μm respectively. In fact, between these windows, light absorption due to the vibrations of chemical molecules (H_2O, CO_2, O_3) present in the atmosphere occur. Nevertheless, as shown in Fig. 1, these glasses are partially transparent in the visible range in the case of glasses based on sulfur or even completely opaque in the case of glasses based on selenium or tellurium. Although it is relatively simple to extend the transmission range to the long wavelengths by adding more massive chalcogen elements modifying the phonon vibrations, the extension of the transparency window for the short wavelengths appears much more complex because it is linked to the variation of the electronic band gap. It is then important to understand the microstructure of the glasses in order to influence the gap between the valence band and the conduction band.

Whatever the wavelength, imagery using visible, near-infrared (1.5–2.7 μm) and thermal-infrared (3–5 μm and 8–12 μm) radiations finds a large number of applications still in strong growth. By way of example, for the driving assistance of a vehicle, the visible/near-infrared image makes it possible to better read the road signs and to detect the presence of ice sheet, while thermal imaging allows one to see further in the fog and detect the presence of pedestrians even in the dark

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